

# Epoxidized soybean oil as a potential source of high-temperature lubricants<sup>☆</sup>

A. Adhvaryu<sup>a,b</sup>, S.Z. Erhan<sup>b,\*</sup>

<sup>a</sup> Department of Chemical Engineering, Pennsylvania State University, University Park, PA 16802, USA

<sup>b</sup> Food and Industrial Oil Research, USDA/NCAUR/ARS, University St. Peoria, 1815 N, Peoria, IL 61604, USA

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## Abstract

Development and application of biocompatible lubricants are increasing daily as a result of stringent regulations imposed on mineral oil-based lubricants with their non-biodegradable toxic wastes. Before consumer acceptance of vegetable oil-based lubricants, they must overcome certain poor performance characteristics such as thermal and oxidative instabilities. This work demonstrates the improved performance of epoxidized soybean oil (ESBO) over soybean oil (SBO) and genetically modified high oleic soybean oil (HOSBO) in certain high temperature lubricant application. We validated the thermal and deposit forming tendencies of these oils using micro-oxidation and differential scanning calorimetry in conjunction with identification of oxidized products by infrared spectroscopy and also discussed the function of a phenolic antioxidant in these oils. Boundary lubrication properties under high load and low speed were determined and the variations explained based on the structural differences of these vegetable oils. Published by Elsevier Science B.V.

**Keywords:** Vegetable oils; Epoxidized soybean oil; Oxidation; Lubricants; Boundary lubrication

## 1. Introduction

A steady increase in the use of eco-friendly consumer products like lubricants has occurred as a result of strict government regulations and in-

creasing public concern for a pollution-free environment. In North America alone, during the last two decades, several billion dollars have been spent to clean up accidental oil spills. Some mineral oil-based lubricants, due to their inherent toxicity and non-biodegradable nature pose a constant threat to ecology and vast ground water reserves. These concerns have resulted in increasing interest in vegetable oils that can be substituted for mineral oil-based products in certain applications. Vegetable oils are renewable raw materials that possess certain excellent frictional properties e.g. good lubricity, low volatility, high

<sup>☆</sup> Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

\* Corresponding author. Tel.: +1-309-681-6532; fax: +1-309-681-6340.

E-mail address: [erhansz@ncaur.usda.gov](mailto:erhansz@ncaur.usda.gov) (S.Z. Erhan).

viscosity index, solvency for lubricant additives, and easy miscibility with other fluids, etc. However, a high degree of multiple C–C unsaturations in the fatty acid (FA) chain of many vegetable oils causes poor thermal and oxidative stability and confines their use as lubricants to a modest range of temperature. Several reports in the literature (Randles and Wright, 1992; Asadauskas et al., 1996; Wu et al., 2000) claim the use of vegetable oils such as rapeseed oil, sunflower oil as substitutes for petroleum based lubricating oil and synthetic esters. Attempts have been made to improve the oxidative stability by transesterification of trimethylolpropane and rapeseed oil methyl ester (Uosukainen et al., 1998) and by selective hydrogenation of polyunsaturated C=C bonds of the FA chains (Johansson and Lundin, 1979). Reports have discussed the use of epoxidized unsaturated FA as metal working fluids (Watanabe et al., 1988) and epoxy oils as lubricating additives to eliminate corrosion from chlorine containing compounds (Tao et al., 1996). Esters of dicarboxylic acids with branching have been used as lubricants and hydraulic fluids over a wide range of temperature (Kenneth et al., 1977; Kadesch, 1979).

In this paper, we explored the effectiveness of using epoxidized soybean oil (ESBO) in certain high temperature lubricant applications. The ESBO results are compared with soybean oil (SBO) and high oleic soybean oil (HOSBO). Soybean oils with a high oleic content obtained by genetic modification of SBO have shown considerable improvement in thermal and oxidative stability over SBO. This is primarily due to the elimination of multiple conjugated unsaturations in the FA chain of SBO. A significantly higher performance level compared with HOSBO was achieved using ESBO in certain high-temperature lubricant applications.

## 2. Materials and methods

### 2.1. Materials

Soybean oil was obtained from Pioneer High Bred Intl. Inc., Des Moines, IA; high oleic soybean oil from Optimum Inc. (Urbandale, IA) and epox-

idized soybean oil from Elf Atochem, Blooming Prairie, MN. The oils were used as received commercially without any further purification and processing.

### 2.2. Antioxidant

The antioxidant is an alkylated phenolic compound (Product code: Lubrizol™ 7652) from Lubrizol, Wickliffe, OH. It was blended into the vegetable oils at 40 °C in various concentrations (0.5, 1.0, 1.5 and 2.0 wt.%).

### 2.3. Thin film micro oxidation (TFMO)

A small amount of oil (25 µl) was oxidized as a thin film on a freshly polished high carbon steel catalyst surface with a steady flow (20 cm<sup>3</sup> min<sup>−1</sup>) of dry air. Oxidation tests were done at various temperatures (175, 200, 225, 250 and 275 °C) and time lengths (30, 60, 90, 120 and 150 min) inside a bottomless glass reactor. The temperature was maintained at ±1 °C with a heated aluminum slab placed on top of a hot plate. This arrangement eliminates temperature gradient across the aluminum surface and transferred heat to the catalysts placed on the slab. The constant airflow ensured removal of volatile oxidation products. The test was designed to eliminate any gas diffusion limitation.

After oxidation, the catalyst containing the oxidized oil sample was removed from the oxidation chamber and cooled rapidly under a steady flow of dry N<sub>2</sub> and transferred to a desiccator for temperature equilibration. After approximately 2 h, the catalyst containing the oxidized oil was weighed to determine the volatile loss (or gain) due to oxidation and then soaked (30 min) with tetrahydrofuran (THF) to dissolve the soluble portion of the oxidized oil. After dissolving the soluble portion of the oxidized oil, the catalyst was dried and weighed to determine the remaining insoluble deposit.

### 2.4. Pressurized differential scanning calorimetry (PDSC)

The experiments were carried out using a PC controlled DSC 2910 thermal analyzer from TA

Instruments (New Castle, USA). The instrument has a maximum sensitivity of  $5 \text{ mV cm}^{-1}$  and temperature sensitivity of  $0.2 \text{ mV cm}^{-1}$ . A 1.5–2.0 mg sample was placed in a hermetically sealed type aluminum pan with a pinhole lid for interaction of the sample with the reactant gas (dry air). The controlled diffusion of the gas through the hole greatly restricts the volatilization of the oil while still allowing for saturation of the liquid phase with air. A film thickness of less than 1 mm was required to ensure proper oil–air interaction and to eliminate any discrepancy in the result due to gas diffusion limitations. The module was first temperature calibrated using the melting point of indium metal ( $156.6^\circ\text{C}$ ) at  $10^\circ\text{C min}^{-1}$  heating rate. Dry air was pressurized in the module at a constant pressure of 3450 kPa and a scanning rate of  $10^\circ\text{C min}^{-1}$  was used throughout the experiment. The peak height ( $T_p$ ) and onset ( $T_o$ ) temperatures were calculated from the exotherm in each case. Induction time ( $I_t$ ) was measured for oils containing different additive concentrations (an alkylated phenolic antioxidant; 0.5–2.0 wt.%) using an isothermal scanning rate.

### 2.5. Nuclear magnetic resonance (NMR)

Quantitative  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker ARX-400 spectrometer (Bruker, Rheinstetten, Germany) at frequencies of 400 and 100 MHz, respectively using a 5 mm dual probe. For the  $^1\text{H}$  and  $^{13}\text{C}$  experiments, sample solutions were prepared in deuterated chloroform ( $\text{CDCl}_3$ , 99.8% D) in 15 and 30% v/v concentrations, respectively. Proton NMR spectra were obtained on 16 repeated scans using a  $30^\circ$  pulse angle, pulse width of  $12.7 \mu\text{s}$ , pulse interval of 1 s and acquisition time of 6.8 s. For the distortionless enhancement by polarization transfer (DEPT 135) measurements, similar sample concentration as the  $^{13}\text{C}$  experiment was maintained. This technique was used to identify and compute the relative distribution of  $\text{CH}_n$  ( $n = 0\text{--}3$ ) carbons in the oils.

### 2.6. Fourier transformed infrared spectroscopy (FTIR)

Infrared spectra were recorded on a Perkin–

Elmer Spectrum RX FT-IR system (Beaconsfield, England) equipped with a KBr beam splitter. A regular scanning range of  $400\text{--}4000 \text{ cm}^{-1}$  was used for 16 repeated scans at a spectral resolution of  $4 \text{ cm}^{-1}$  in thin film condition using a pair of KBr crystals. All the spectra were recorded and processed using the Perkin–Elmer data processing software.

### 2.7. Friction measurement by ball-on-disk method

Boundary lubrication properties of SBO, HOSBO and ESBO were studied using a multi-specimen friction measurement apparatus of FALEX (Sugar Grove, IL). Ball-on-disk experiments (1018 steel disk, Rc 15–25) were carried out under low speed  $6.22 \text{ mm s}^{-1}$  (5 rpm) and high load 181.44 kg at  $25^\circ\text{C}$  using test oils diluted to different concentrations with hexadecane. Measurements of coefficient of friction (CoF) and torque were made in each case. The CoF values reported are averages of two or three independent experiments and the standard deviation observed was  $\pm 0.02$ .

## 3. Results and discussion

The physicochemical properties of SBO, HOSBO and ESBO are presented in Table 1. The viscosity of ESBO at  $40^\circ\text{C}$  is significantly larger than the other oils. The higher molecular weight and more polar structure compared with SBO and HOSBO results in stronger intermolecular interaction in ESBO. This property of ESBO would translate into enhanced lubricity in a dynamic system. The data indicate that nearly entire  $\text{C}=\text{C}$  unsaturation in FA chain have been converted to epoxy group. These properties will cumulatively influence the thermal and oxidative behavior of ESBO.

A partial proton NMR spectrum of ESBO is shown in Fig. 1. The  $\text{C}=\text{C}$  unsaturation present in SBO (normally at 5.2 ppm) is largely replaced with the epoxy group of ESBO. The peak assignments were made using DEPT 135 and COSY 45 NMR experiments. The epoxy protons are observed in the  $\delta$  3.0–3.2 ppm region. The methine

Table 1  
The physical properties of vegetable oils

Properties	Vegetable oils		
	SBO	HOSBO	ESBO
Appearance	Light yellow	Pale yellow	Colorless
Kinematic viscosity, cSt (ASTM D445) <sup>a</sup> at 40 °C	32.93	41.34	170.85
Kinematic viscosity, cSt (ASTM D445) <sup>a</sup> at 100 °C	8.08	9.02	20.41
Acid value (mg KOH g <sup>-1</sup> ) (AOCS, Ca 5a-40) <sup>b</sup>	0.16	0.12	0.09
Peroxide value (meq. kg <sup>-1</sup> ) (AOCS, Cd 8-53) <sup>b</sup>	9.76	4.78	0.0
Iodine value (mg I <sub>2</sub> g <sup>-1</sup> ) (AOCS, Cd 1-25) <sup>b</sup>	144.8	85.9	9.11
Fatty acid composition by GC (AACC, 58-18) <sup>c</sup> in %			
C <sub>16:0</sub>	6.0	6.0	70.0
C <sub>18:0</sub>	5.5	3.0	30.0 <sup>d</sup>
C <sub>18:1</sub>	22.0	85.0	0.0
C <sub>18:2</sub>	66.0	4.0	0.0
C <sub>18:3</sub>	0.5	2.0	0.0

<sup>a</sup> ASTM D-445, annual book of ASTM standards, vol. 05.01, 2000.

<sup>b</sup> Official methods and recommended practices of the AOCS, 5th edition, 1998.

<sup>c</sup> Approved methods of the American Association of Cereal Chemists, 10th edition, vol. II, 2000.

<sup>d</sup> Obtained from the epoxidation of C<sub>18:1</sub>, C<sub>18:2</sub> and C<sub>18:3</sub>.

proton of  $-\text{CH}_2-\text{CH}-\text{CH}_2-$  backbone at  $\delta$  5.1–5.3 ppm, methylene proton of  $\text{CH}_2-\text{CH}-\text{CH}_2$  backbone at  $\delta$  4.0–4.4 ppm,  $\text{CH}_2$  proton adjacent to two epoxy group at  $\delta$  2.8–3.0 ppm,  $-\text{CH}-$  protons of the epoxy ring at  $\delta$  3.0–3.2 ppm,  $\alpha\text{-CH}_2$  to  $>\text{C}=\text{O}$  at  $\delta$  2.2–2.4 ppm,  $\alpha\text{-CH}_2$  to epoxy group at 1.7–1.9 ppm,  $\beta\text{-CH}_2$  to  $>\text{C}=\text{O}$  at  $\delta$  1.55–1.7 ppm,  $\beta\text{-CH}_2$  to epoxy group at  $\delta$  1.4–1.55 ppm, saturated methylene groups at  $\delta$  1.1–1.4 ppm and terminal  $-\text{CH}_3$  groups at  $\delta$  0.8–1.0 ppm region. The olefin and bisallylic protons in SBO and HOSBO have high susceptibility to thermal and oxidative degradation. The protons associated with these carbon atoms are highly labile and trigger radical initiated oxidation in the presence of oxygen at these sites. The removal of these reactive protons in ESBO would suggest an enhanced oxidative stability in these oils.

ESBO shows a characteristic peak at  $823\text{ cm}^{-1}$  from the epoxy group (Fig. 2). The other peaks observed from the partial FTIR spectra are: 723 (methylene in-phase rocking), 957, 1017, 1105 (ether, antisymmetric stretch), 1163, 1239 (ester, antisymmetric stretch), 1378 (methyl symmetric

deformation), 1463 (methyl antisymmetric deformation) and 1745 (esters, aliphatic  $\text{C}=\text{O}$  stretch)  $\text{cm}^{-1}$ . The spectra obtained for SBO and HOSBO are similar to ESBO except for the absorption due to the epoxy group.

The deposit values obtained from thin film micro oxidation (TFMO) at 175 °C at different time intervals are shown in Fig. 3. The tests, carried out in static mode under a steady flow of air ( $20\text{ ml min}^{-1}$ ) in a bottomless glass reactor, were terminated and analyzed at the indicated times. After the stipulated test time, the oxidized

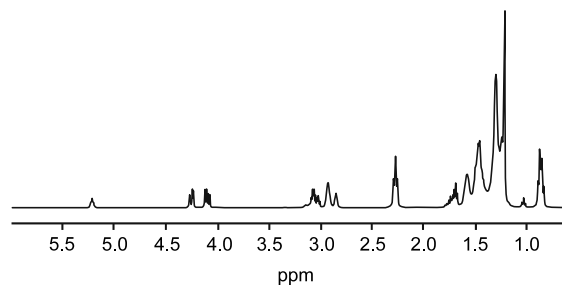


Fig. 1. Partial  $^1\text{H}$  NMR spectra of epoxidized soybean oil (ESBO).

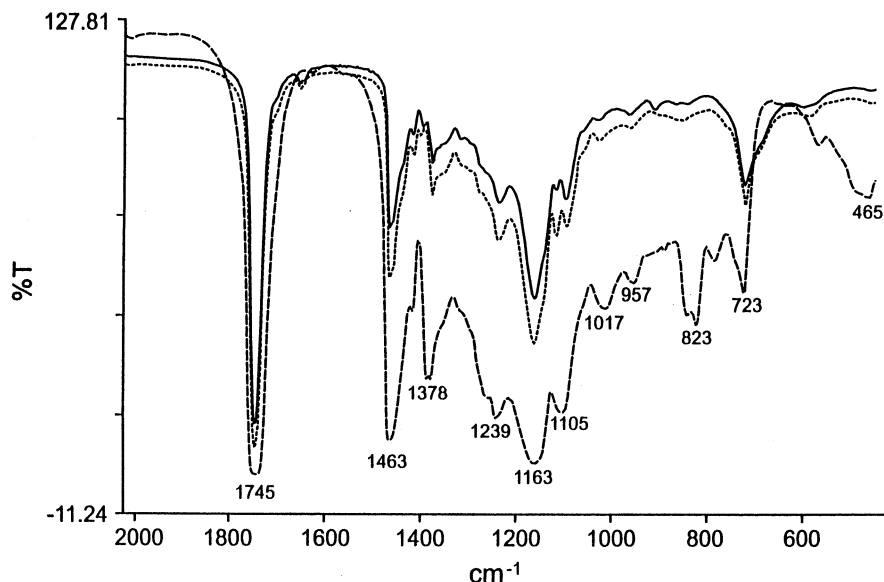


Fig. 2. Overlay infrared transmittance spectra of SBO (—); HOSBO (···) and ESBO (---) showing the position of major peaks.

oil was washed with THF and the residue left on the coupon is termed as insoluble deposit. During the oxidation process, several primary oxidation products are formed, which later in the presence of excess  $O_2$  undergo further oxy-polymerization reaction to form oil insoluble deposit (Adhvaryu et al., 2000). The tendency to form such deposit is the main detrimental factor associated with vegetable oil used as high-temperature lubricants. The relative influence of various structural parameters of vegetable oils during oxidation is described in a separate study (Adhvaryu et al., 2000). The data of Fig. 3 shows that ESBO remains fairly stable. No significant oxidative degradation occurs until after 60 min into the oxidation. The following sharp increase in the deposit formation suggests a rapid breakdown of the epoxy group leading to oxidative polymerization through oxygen bonding. The induction time for deposit formation of ESBO is roughly twice that of HOSBO under similar oxidation temperature (175 °C). The percent insoluble deposit becomes stable after 2 h of oxidation with ESBO remaining lower compared with other oils. Larger insoluble deposit observed in SBO and HOSBO at different time periods compared with ESBO, is

mainly due to their linoleic and oleic content in the FA chains (see Table 1). SBO shows greater deposit primarily than HOSBO due to the presence of conjugated unsaturation in the FA chain relative to HOSBO.

The partial FTIR spectra of the carbonyl region of oxidized SBO, HOSBO and ESBO, after 90 min test shows that such oxidation process results in the formation of a broad carbonyl envelop in the FTIR spectra due to an array of closely spaced oxygenated carbonyl compounds

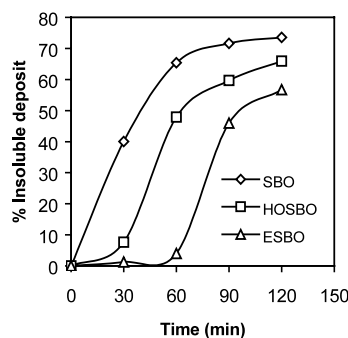


Fig. 3. Percent insoluble deposit of SBO, HOSBO and ESBO obtained from TFMO at 175 °C under air flow (20 ml min<sup>-1</sup>); sample size = 25 µl.

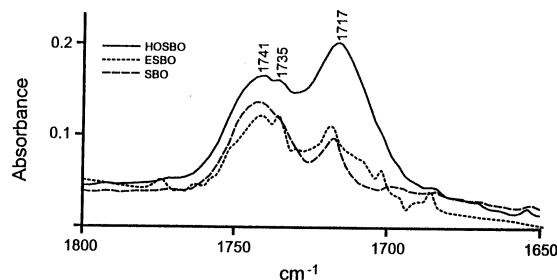


Fig. 4. Partial FTIR absorbance spectra of oxidized SBO, HOSBO and ESBO showing the position of different functional groups in the carbonyl region.

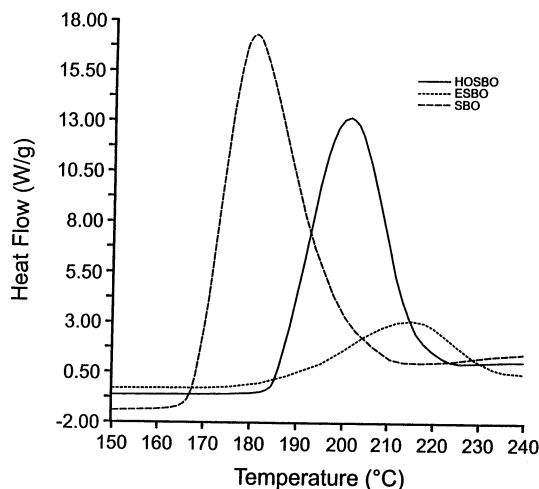


Fig. 5. Exothermic plots of SBO, HOSBO and ESBO at a scanning rate of  $10\text{ }^{\circ}\text{C min}^{-1}$  under constant pressure (3450 kPa).

(Fig. 4). In the spectral range of  $1800\text{--}1650\text{ cm}^{-1}$ , three characteristic absorbance peaks are present at  $1717$ ,  $1735$  and  $1741\text{ cm}^{-1}$ . The ab-

sorption peak at  $1717\text{ cm}^{-1}$  due to ketone functionality is prominent in all the oxidized oils with varying intensity. Another peak at  $1735\text{ cm}^{-1}$  from cyclic lactones (mainly 6 member) makes a shoulder with the absorption peak at  $1741\text{ cm}^{-1}$  from peroxy ester groups. The formation of these oxy-compounds is due to radical initiated oxidation reaction in presence of  $\text{O}_2$ . Several other studies have reported the formation of cyclic structures during oxidation (Naidu et al., 1984; Adhvaryu et al., 1998). These compounds form the precursors of high temperature polymeric deposits associated with vegetable oil oxidation.

PDSC is an effective way of measuring the oxidative tendency of vegetable oils in an accelerated mode. At high air pressure (3450 kPa), the concentration of  $\text{O}_2$  is in excess and at equilibrium with the sample. Thus, any inconsistency due to access rate of oxygen and egress rate of volatile degradation product is effectively eliminated. The exothermic plot of SBO, HOSBO and ESBO at a scanning rate of  $10\text{ }^{\circ}\text{C min}^{-1}$  under constant pressure is shown in Fig. 5. The peak temperature ( $T_p$ ) and onset temperature ( $T_o$ ) combination of these oils are SBO =  $180.3/167.8$ , HOSBO =  $201.1/185.8$  and ESBO =  $214.9/188.1\text{ }^{\circ}\text{C}$ . The ESBO shows high oxidative stability compared with SBO and HOSBO. The TFMO and FTIR data also show similar results.

The effects of an antioxidant additive on the  $T_p$  and  $T_o$  of vegetable oils are listed in Table 2. ESBO showed maximum improvement in oxidative stability at the lower additive concentration (0.5 wt.%) followed by HOSBO and SBO. A plateau was reached at higher additive concentration (1.5 wt.%) and no concentration beyond this had any improvement in oxidation stability.

Table 2

PDSC of vegetable oils at different antioxidant concentrations: scanning rate =  $10\text{ }^{\circ}\text{C min}^{-1}$ , constant pressure = 2450 kPa

Vegetable oils	0.5% AO		1.0% AO		1.5% AO		2.0% AO	
	$T_p$ ( $^{\circ}\text{C}$ )	$T_o$ ( $^{\circ}\text{C}$ )	$T_p$ ( $^{\circ}\text{C}$ )	$T_o$ ( $^{\circ}\text{C}$ )	$T_p$ ( $^{\circ}\text{C}$ )	$T_o$ ( $^{\circ}\text{C}$ )	$T_p$ ( $^{\circ}\text{C}$ )	$T_o$ ( $^{\circ}\text{C}$ )
SBO	190.3	178.9	194.3	183.6	197.8	188.5	199.6	190.8
HOSBO	213.6	203.3	220.5	211.7	225.6	217.2	229.9	221.7
ESBO	251.3	243.3	259.9	252.3	262.9	254.9	264.0	256.0

$T_p$  and  $T_o$  values are averages of three independent experiments. Standard error =  $\pm 1\text{ }^{\circ}\text{C}$ .

Table 3

Oxidation induction time ( $I_t$ ) (isothermal condition) of vegetable oils at different antioxidant additive concentrations: constant pressure = 2450 kPa

	0.5% AO time (min)	1.0% AO time (min)	1.5% AO time (min)	2.0% AO time (min)
SBO (150 °C) <sup>a</sup>	7.84 ± 0.6	12.81 ± 0.7	17.44 ± 0.5	23.03 ± 0.8
HOSBO (160 °C) <sup>a</sup>	28.74 ± 0.8	57.35 ± 1.3	88.93 ± 1.7	127.12 ± 2.2
ESBO (195 °C) <sup>a</sup>	52.24 ± 1.0	76.84 ± 1.8	86.47 ± 1.5	89.85 ± 1.6

AO, antioxidant (an alkylated phenolic compound);  $I_t$  values are averages of three independent experiments.

<sup>a</sup> Isothermal temperature.

Similarly, for oxidation induction time ( $I_t$ ) measurement under isothermal condition (Table 3), ESBO (at 195 °C) showed greater stability as compared with SBO (at 150 °C) and HOSBO (at 160 °C). The induction time data shows that the isothermal temperature for the oxidation of ESBO is significantly higher than HOSBO. The ESBO is run at an isothermal temperature 35 °C higher than the HOSBO. This suggests stability significantly higher for ESBO than HOSBO by a factor of 6 or more. The trend is in agreement with the deposit forming tendencies (Fig. 3). PDSC therefore can, be used effectively to optimize the additive concentration in vegetable oil blends and rapidly assay their thermal and oxidative behavior.

Vegetable oils are known to provide excellent lubricity due to their ester functionality (Stern, 1989). The polar heads of the fatty acid chains attach to metal surfaces by a chemical process that allows a monolayer film formation with the non-polar end of fatty acids sticking away from the metal surface. The fatty acid  $-\text{CH}_2-$  chain offers a sliding surface that prevents the participating metal from making direct contact with each other. When a film is not formed, contact may result in rising temperature at the contact zones of moving parts causing adhesion, scuffing or even metal-to-metal welding. During this rubbing process under lubricated condition, at high load and low speed, bond cleavage of fatty acid molecule might take place. Under such condition, the epoxy group of the ESBO offers active oxygen sites that trigger polymerization on the metal surface forming a protective film. This protective film builds further with time to reduce friction. The CoF data of ball-on-disk experiment of veg-

etable oils blended into hexadecane in different molar concentration is shown in Table 4. Under a high load of 181.44 kg and low speed 6.22 mm s<sup>-1</sup> (5 rpm), all of the vegetable oils show excellent reduction in CoF at low molar concentration.

#### 4. Conclusions

This study offers a detailed insight about the thermal, oxidative and frictional behavior of ESBO as a potential candidate for high temperature lubrication. The deposit-forming tendency of ESBO is significantly reduced due to the removal of multiple unsaturations in fatty acid chains. ESBO shows good response to antioxidant additives at low concentration (1%) and maintains a

Table 4

Coefficient of friction values of vegetable oils in different concentrations of hexadecane

Concentration (M)	Coefficient of friction		
	SBO	HOSBO	ESBO
0.003	0.269	0.248	0.232
0.004	0.243	0.221	0.220
0.005	0.223	0.205	0.171
0.007	0.203	0.190	0.165
0.01	0.171	0.163	0.144
0.03	0.135	0.132	0.104
0.1	0.095	0.108	0.102
0.2	0.092	0.100	0.102
0.4	0.092	0.100	0.100
0.6	0.090	0.090	0.100

Load = 181.44 kg and speed = 6.22 mm min<sup>-1</sup> at 25 °C. CoF values are averages of two or three independent experiments. Standard deviation = ± 0.02.

low thermal and oxidative behavior compared with other test oils. Further, ESBO, SBO and HOSBO have excellent coefficient of friction reducing properties due to the formation of a stable polymeric film on the metal surface during boundary lubrication. Such film formation in ESBO is achieved through the –O– cross linking on the metal surface, and thus reducing friction between the moving parts.

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